

Stress Corrosion Cracking of High Strength Steels

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ABSTRACT

The stress corrosion cracking (SCC) was investigated for AerMet 100 and 300M steels in four aqueous NaCl solutions of different concentrations (0.035-3.5%), three of which had an identical electrical conductivity ($12.44 \times 10^{-4} \text{ m}^2 \text{ S mol}^{-1}$). Especially, the variation of threshold stress intensity for SCC, K_{ISCC} , with cathodic potential was evaluated, employing rising step load test method. The K_{ISCC} increased, peaked at around the potential of -0.7 V_{SCE} , and then decreased with increasing potential for AerMet 100 steel. On the other hand, the K_{ISCC} did not change much with potential for 300M steel. The open circuit potential and the corresponding K_{ISCC} were greater for AerMet 100 steel than for 300M steel, indicating the former nobler and more SCC resistant. The SEM fractographs showed mixed cleavage and intergranular cracking, more cleavage for AerMet 100 steel but more intergranular for 300M steel, at all potentials employed.

INTRODUCTION

Because of its high ultimate tensile strength (UTS), 2,000 MPa, 300M steel had been accepted as the material for aircraft landing gear and other structural components. In 1990, a new steel, AerMet 100, was developed. It has a good combination of high UTS, 1,979 MPa, and high fracture toughness, $126 \text{ MPa}\sqrt{\text{m}}$, which is much greater than that of 300M steel, $57 \text{ MPa}\sqrt{\text{m}}$. Consequently, AerMet 100 steel can replace 300M steel to increase damage tolerance. On the one hand, the two steels are susceptible to corrosion, and protective plating is required for service in corrosive environments.

The SCC of AerMet 100 and 300M steels had been investigated rather extensively, employing various test methods: cantilever bend¹, double cantilever beam² and rising step load³. However, the results had not been quite consistent, as evidenced by different values¹⁻³ of K_{ISCC} , shown in Table 1. Furthermore, the test environment had been limited to an aqueous 3.5% NaCl solution. Therefore, this study was initiated to identify the effect of NaCl concentration on SCC, and also evaluate and compare the SCC resistance of AerMet 100 and 300M steels in aqueous solutions of different NaCl concentrations.

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| <u>Test Method</u> | <u>K_{ISCC} (MPa√m)</u> | |
|---|---------------------------------|-------------|
| | <u>AerMet</u> | <u>300M</u> |
| Cantilever Bend ¹ (10,000 Hours) | 17-24 | 12-15 |
| Double Cantilever ² Beam | 36 | 21 |
| Rising Step Load ³ (Open Circuit Potential) | 28 | |

Table 1. K_{ISCC} in Aqueous 3.5% NaCl Solution

EXPERIMENTAL PROCEDURE

The specimen materials were a forged AerMet 100 steel slab of 2.5 x 10 x 122 cm and a forged 300M steel rod of 13 cm diameter and 51 cm length. Their chemical compositions are shown in Table 2.

| <u>Element</u> | <u>AerMet</u> | <u>300M</u> |
|----------------|---------------|-------------|
| C | 0.23 | 0.43 |
| Ni | 11.12 | 1.80 |
| Cr | 2.99 | 0.80 |
| Mo | 1.17 | 0.40 |
| Co | 13.44 | - |
| Mn | 0.01 | 0.75 |
| Si | 0.02 | 1.60 |
| P | 0.002 | 0.005 |
| S | 0.0005 | 0.005 |
| Fe | balance | balance |

Table 2. Chemical Compositions (%) of AerMet 100 and 300M Steels

These steels were subjected to the following heat treatment, respectively.

| <u>Steel</u> | <u>Solution Treatment</u> | <u>Quenching in</u> | <u>Freezing</u> | <u>Aging in</u> |
|--------------|---------------------------|---------------------|-----------------|----------------------|
| | <u>in Vacuum</u> | | | <u>Vacuum</u> |
| AerMet | 885°C, 1.25 Hr | N ₂ Gas | -73°C, 2 Hr | 482°C, 5 Hr |
| 300M | 871°C, 5 Hr | Oil | - | 302°C, 2 Hr (Double) |

Table 3. Heat Treatment for AerMet 100 and 300M Steels

The heat treatment resulted in hardness R_c 56 and 53 and microstructures, shown in Figure 1, for the AerMet 100 and 300M steels, respectively. Subsequently, v-notch (60°) square bar specimens of the two steels were machined, Figure 2.

Since the cantilever bend and double cantilever beam tests require a long time, an accelerated test method of rising step load⁴ was employed. The specimen was fatigue-precracked to $\frac{1}{2}$ thickness, and then step-loaded in four-point bending under constant displacement control at room temperature, while held at a predetermined potential in a corrosive electrolyte. The load was increased at 2% of the UTS each hour until load drop, which corresponds to crack initiation. A schematic of the rising step loading is shown in Figure 3. The stress intensity factor was calculated as a function of applied bending moment and crack length, using the following equation.

$$K = \sigma \sqrt{\pi a} F(a/W)$$

where

K: stress intensity factor

σ : gross stress = $6M/bW^2$

M: bending moment = Px

P: applied load

x: moment arm length

b: specimen thickness

W: specimen width

a: crack length

$$F(a/W) = 1.122 - 1.40(a/W) + 7.33(a/W)^2 - 13.08(a/W)^3 + 14.0(a/W)^4$$

As the corrosive electrolytes, aqueous solutions of four different NaCl and Na₂SO₄ concentrations were used. Three of them had an identical electrical conductivity, $126.44 \times 10^{-4} \text{ m}^2 \text{ S mol}^{-1}$. They were

| No. | Aqueous Solution (%) | | pH | Electrical Conductivity |
|-----|----------------------|---------------------------------|-----|--|
| | NaCl | Na ₂ SO ₄ | | $10^{-4} \text{ m}^2 \text{ S mol}^{-1}$ |
| 1 | 3.5 | 0.0 | 7.3 | - |
| 2 | 3.5 | 2.8 | 6.3 | 126.44 |
| 3 | 0.35 | 6.5 | 6.1 | 126.44 |
| 4 | 0.035 | 6.9 | 6.1 | 126.44 |

Table 4. Aqueous NaCl Solutions of Various Concentrations

Prior to the rising step load test, the open circuit or free corrosion potential was measured for each steel in each solution.

During the test, the specimen was the working electrode in a three-electrode cell with a saturated calomel reference electrode and a platinum counterelectrode. Cathodic potentials, ranging from $-1.2 \text{ V}_{\text{SCE}}$ (volts versus a saturated calomel electrode) to the respective open circuit potential, were applied to generate various amounts of hydrogen in front of the advancing crack tip.

After the SCC test, the fracture surface morphology was examined with a scanning electron microscope (SEM), JEOL JSM-5800LV, operating at a 20 kV accelerating voltage.

RESULTS

1. Open Circuit Potentials

The open circuit potentials (OCP) for the two steels in the four solutions were determined to be

| No. | Aqueous Solution (%) | | OCP (V_{SCE}) | |
|-----|----------------------|---------------------------------|-------------------|-------|
| | NaCl | Na ₂ SO ₄ | AerMet | 300M |
| 1 | 3.5 | 0.0 | -0.51 | -0.68 |
| 2 | 3.5 | 2.8 | -0.56 | -0.70 |
| 3 | 0.35 | 6.5 | -0.61 | -0.72 |
| 4 | 0.035 | 6.9 | -0.61 | -0.71 |

Table 5. Open Circuit Potentials

2. Test in Aqueous 3.5% NaCl Solution (No, 1)

The variation of K_{ISCC} with potential is illustrated for the two steels, tested in the aqueous 3.5% NaCl solution (No. 1), in Figure 4. At more negative potentials, the K_{ISCC} values of the two steels tend to close together, and they are 14.2 and 15.9 MPa \sqrt{m} at $-1.2 V_{SCE}$ for the AerMet 100 and 300M steels, respectively. However, with increasing potential, the K_{ISCC} increases, peaks at -0.7 and $-0.6 V_{SCE}$, and then decreases for AerMet 100 steel. On the other hand, it is relatively constant, 15.9-17.5 MPa \sqrt{m} , at all potentials, ranging from -1.2 to $-0.68 V_{SCE}$, for 300M steel.

The color of electrolyte changed from transparent below $-0.8 V_{SCE}$ to rusty red above $-0.8 V_{SCE}$, indicating a dissolution reaction, for the both steels.

The K_{ISCC} values at the open circuit potentials, -0.51 and $-0.68 V_{SCE}$, were found to be 25.6 and 17.5 MPa \sqrt{m} for the AerMet 100 and 300M steels, respectively.

The SEM fractograph of the AerMet 100 steel shows a mixture of more cleavage and interspersed intergranular cracking without any appreciable change across the all potentials employed, Figure 5. Namely, the potential change from -1.2 to $-0.51 V_{SCE}$ changes the SCC resistance, K_{ISCC} , but does not change its mode.

The SEM fractograph of the 300M steel shows a mixture of somewhat more intergranular and interspersed cleavage cracking without any noticeable change across the all potentials employed, Figure 6. Compared to the AerMet 100 steel, the 300M steel has larger grains of prior austenite. The constant cracking mode is consistent with the relatively constant values of K_{ISCC} across the all potentials employed.

3. Test in Aqueous NaCl and Na₂SO₄ Solutions of Identical Electrical Conductivity (No. 2– 4)

Figures 7-9 show the variation of K_{ISCC} with potential for the two steels in the three aqueous solutions of different concentrations but an identical electrical conductivity: 3.5% NaCl and 2.8% Na₂SO₄, 0.35% NaCl and 6.5% Na₂SO₄, and 0.035% NaCl and 6.9% Na₂SO₄. For the two steels, the K_{ISCC} values range from 12.1 to 16.0 MPa√m at $-1.2 V_{SCE}$. For AerMet 100 steel, the K_{ISCC} increases, peaks at $-0.7 V_{SCE}$, and then decreases with increasing potential. On the other hand, the K_{ISCC} value of 300M steel does not change much with potential, ranging from 14.5 to 17.7 MPa√m.

The K_{ISCC} values of the two steels at the open circuit potentials in the three solutions were determined to be

| No. | Aqueous Solution (%) | | OCP (V_{SCE}) | | K_{ISCC} (MPa√m) | |
|-----|----------------------|---------------------------------|-------------------|-------|--------------------|------|
| | NaCl | Na ₂ SO ₄ | AerMet | 300M | AerMet | 300M |
| 2 | 3.5 | 2.8 | -0.56 | -0.70 | 32.4 | 16.8 |
| 3 | 0.35 | 6.5 | -0.61 | -0.72 | 36.8 | 16.8 |
| 4 | 0.035 | 6.9 | -0.61 | -0.71 | 36.8 | 16.8 |

Table 6. K_{ISCC} Values of Two Steels at Open Circuit Potentials in Three Solutions of Identical Electrical Conductivity

The SEM fractographic features of the two steels are similar to those for the test in the aqueous 3.5% NaCl solution (No. 1), more cleavage in the AerMet 100 steel and more intergranular in the 300M steel.

DISCUSSION

The observed increase in K_{ISCC} with increasing potential for the AerMet 100 steel is attributable to the accompanying reduction of hydrogen generation at the advancing crack tip and the requirement of a higher stress intensity for crack propagation in the four aqueous solutions of different concentrations. On the other hand, the K_{ISCC} changes a little with increasing potential for the 300M steel in the four aqueous solutions. This evidences that the 300M steel has a higher embrittlement sensitivity to hydrogen content that is saturated at the all potentials, ranging from -1.2 to $-0.68 V_{SCE}$. The comparatively lower embrittlement sensitivity of the AerMet 100 steel to hydrogen is not overcome until near the potential of $-1.2 V_{SCE}$, where the AerMet 100 steel behaves the same as the 300M steel.

The determined open circuit potential is greater for the AerMet 100 steel than for the 300M steel, indicating the former nobler and more corrosion resistant than the latter, in the four aqueous solutions. In addition, the open circuit potential is greater in the 3.5% NaCl solution (No. 1) than in the three aqueous solutions of an identical electrical conductivity (No. 2– 4) for the both steels.

The K_{ISCC} of the AerMet 100 steel at the open circuit potential in the 3.5% NaCl solution, 25.6 MPa \sqrt{m} , is close to that reported by Buckley³, 27.5 MPa \sqrt{m} . On the one hand, it is greater than that for the 300M steel, indicating greater SCC resistance for the AerMet 100 steel than for the 300M steel in the solution. Furthermore, it is less than those in the three aqueous solutions of an identical electrical conductivity for the AerMet 100 steel, whereas it is not much different in all of the four solutions for the 300M steel. This indicates that:

- The AerMet 100 steel is less susceptible to SCC in the three aqueous solutions of an identical electrical conductivity than in the 3.5% NaCl solution (No. 1).
- With an identical electrical conductivity, the change in NaCl and Na₂SO₄ concentration from 0.35 to 0.035% does not change the SCC resistance of the AerMet 100 steel.
- With an identical electrical conductivity, the NaCl and Na₂SO₄ concentration does not affect the SCC resistance of the 300M steel.

The SCC mode was found to be a mixture of more cleavage and interspersed intergranular cracking for AerMet 100 steel and the reverse for 300M steel. The cracking mode did not change noticeably across the all potentials employed for the both steels. The more intergranular SCC mode, observable irrespective of potential, demonstrates a weaker link of the prior austenite grain boundaries in the 300M steel than in the AerMet 100 steel.

In their study on PH 13-8 steel, Tyler, Levy and Raymond⁶ observed subtle change in the fracture surface morphology of dominant cleavage between the -1.2 and -0.8 V_{SCE} region. On the other hand, they observed clear changes in fracture mode from intergranular (-1.2 V_{SCE}) to cleavage (-0.8 V_{SCE}) for T-250 maraging steel.

CONCLUSIONS

1. The K_{ISCC} increases, peaks at around -0.7 V_{SCE}, and then decreases with increasing potential for AerMet 100 steel in the four solutions of different concentrations. However, it does not change much with potential for 300M steel in those solutions.
2. The open circuit potential and the corresponding K_{ISCC} are

| Aqueous Solution (%) | | OCP (V _{SCE}) | | K_{ISCC} (MPa \sqrt{m}) | |
|----------------------|---------------------------------|-------------------------|-------|------------------------------|------|
| NaCl | Na ₂ SO ₄ | AerMet | 300M | AerMet | 300M |
| 3.5 | 0.0 | -0.51 | -0.68 | 25.6 | 17.5 |
| 3.5 | 2.8 | -0.56 | -0.70 | 32.4 | 16.8 |
| 0.35 | 6.5 | -0.61 | -0.72 | 36.8 | 16.8 |
| 0.035 | 6.9 | -0.61 | -0.71 | 36.8 | 16.8 |

The above evidences that

- The AerMet 100 steel has greater SCC resistance than the 300M steel in the four aqueous solutions.

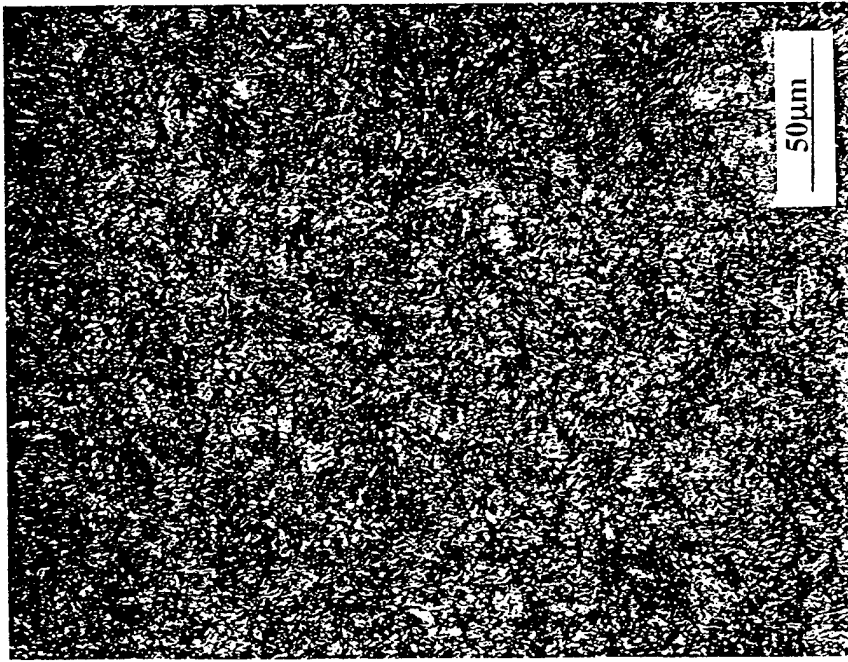
- With an identical electrical conductivity, the change in NaCl and Na₂SO₄ concentration from 0.35 to 0.035% does not change the SCC resistance of the AerMet 100 steel.
 - With an identical electrical conductivity, the NaCl and Na₂SO₄ concentration does not affect the SCC resistance of the 300M steel.
3. SCC mode is a mixture of cleavage and intergranular cracking, more cleavage for the AerMet 100 steel and more intergranular for the 300M steel, and it is independent of potential.

ACKNOWLEDGMENT

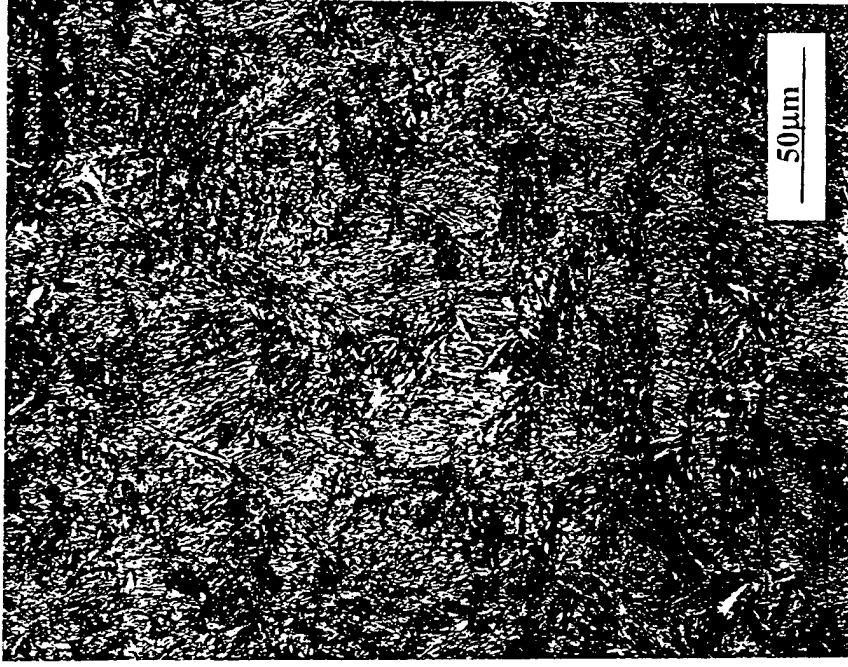
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(a) AerMet 100 Steel



(b) 300M Steel

Figure 1. Microstructures of Specimen Materials

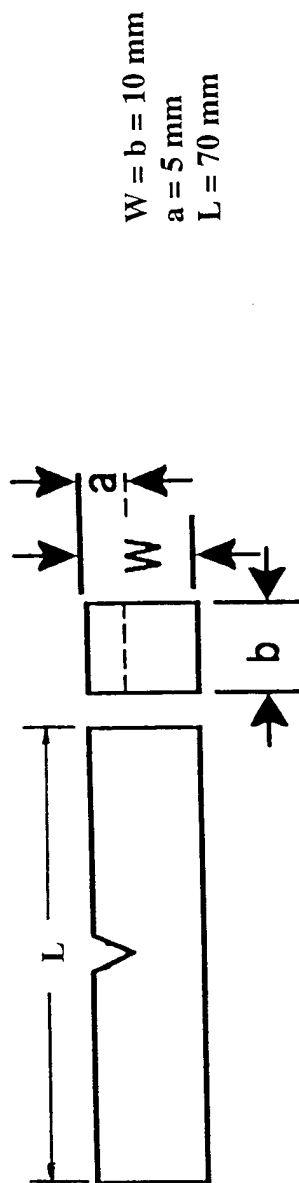


Figure 2. Square Bar Specimen

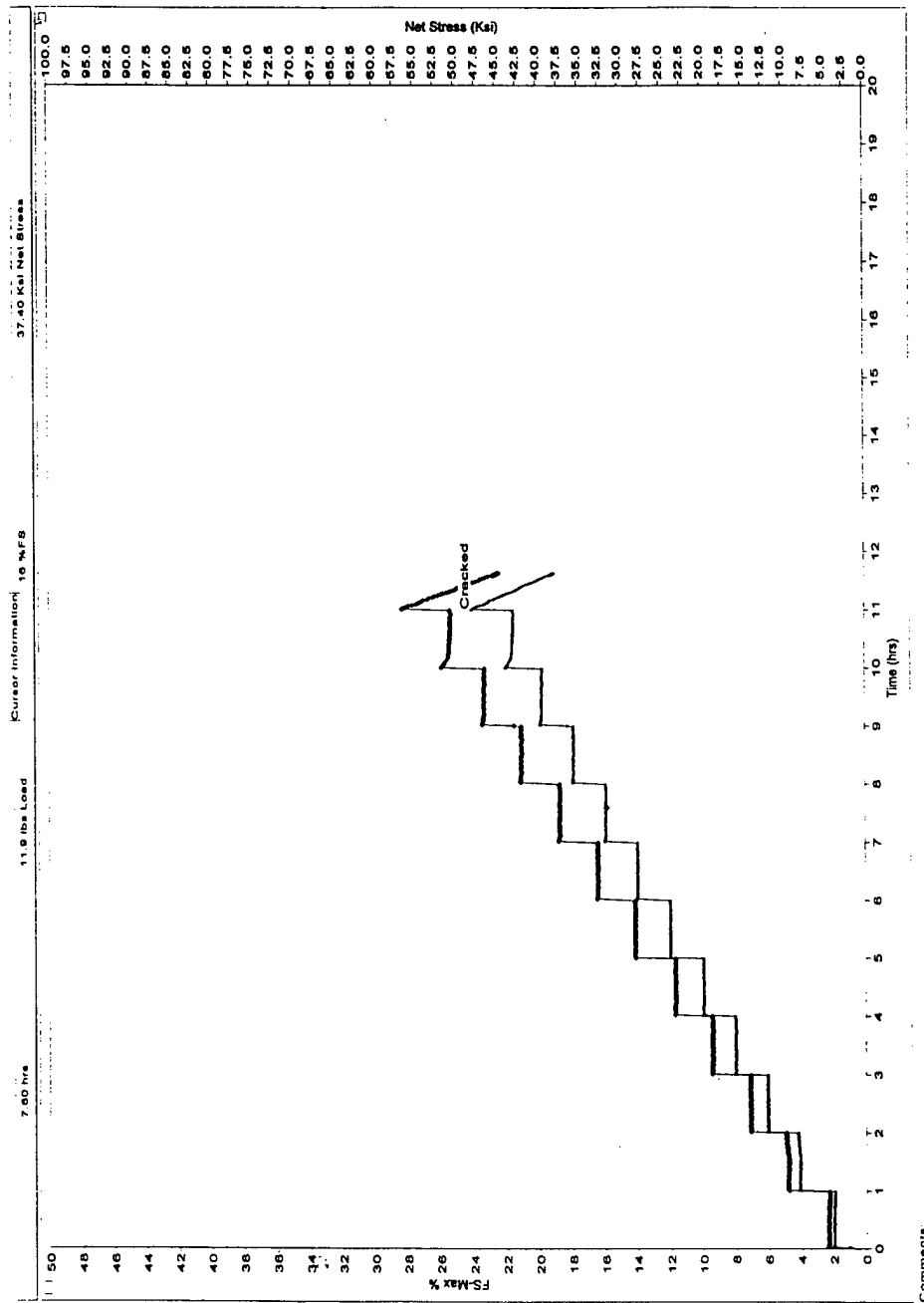


Figure 3. Schematic of Rising Step Loading

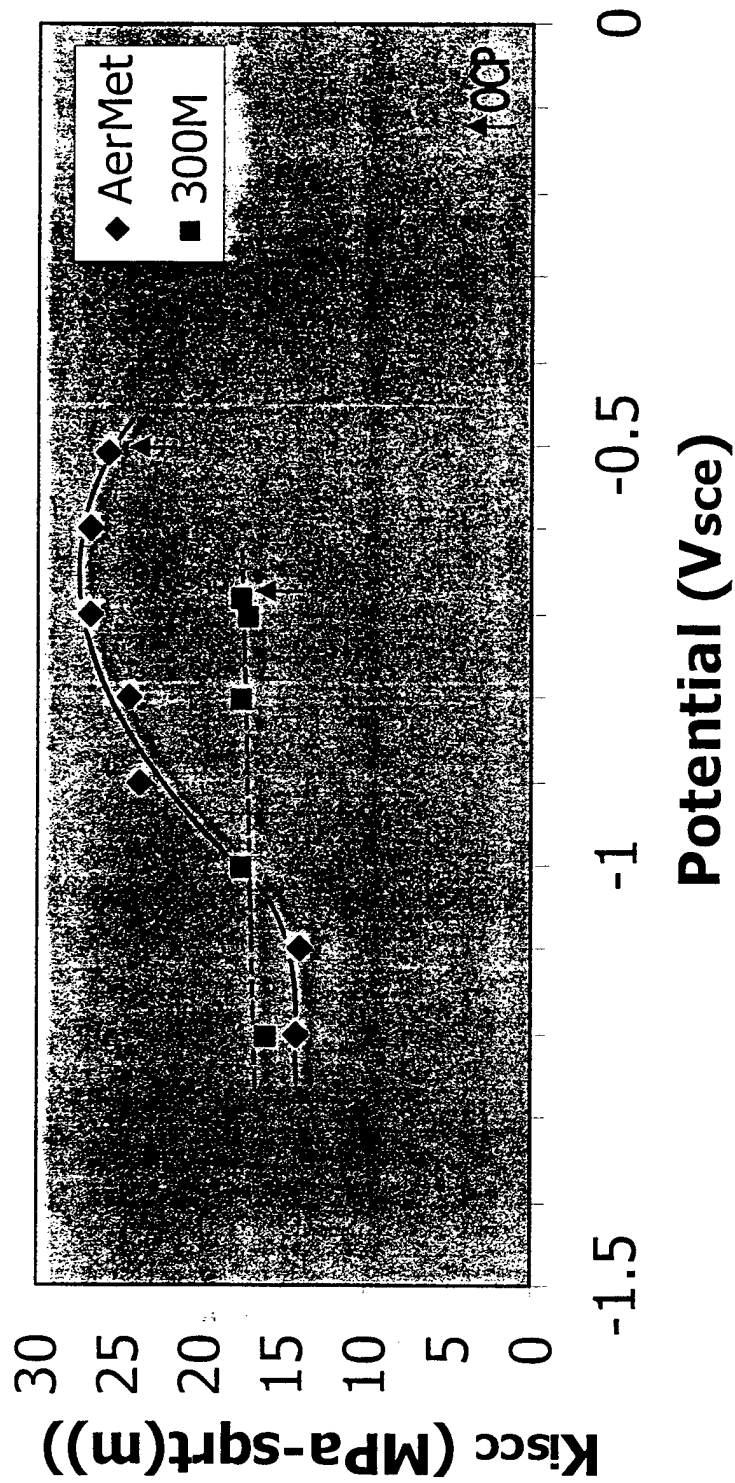
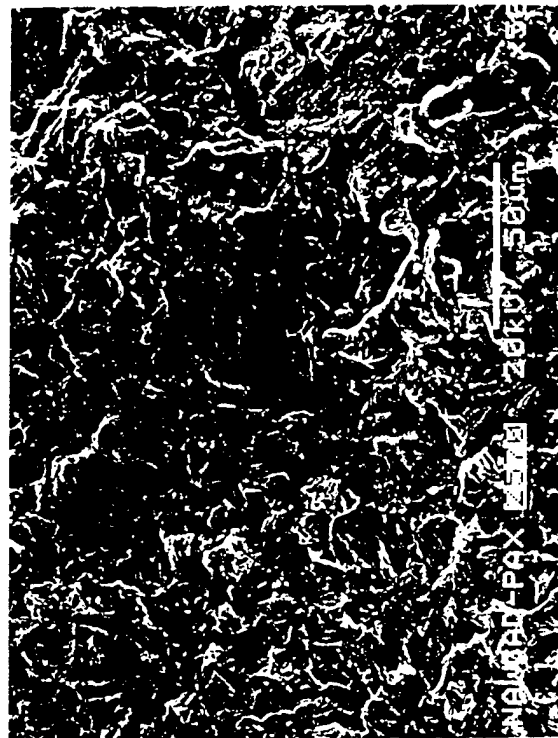


Figure 4. Variation of K_{ISCC} with Potential in Aqueous Solution of 3.5% NaCl



-1.2 V_{SCE}



-1.0 V_{SCE}



-0.8 V_{SCE}



-0.6 V_{SCE}

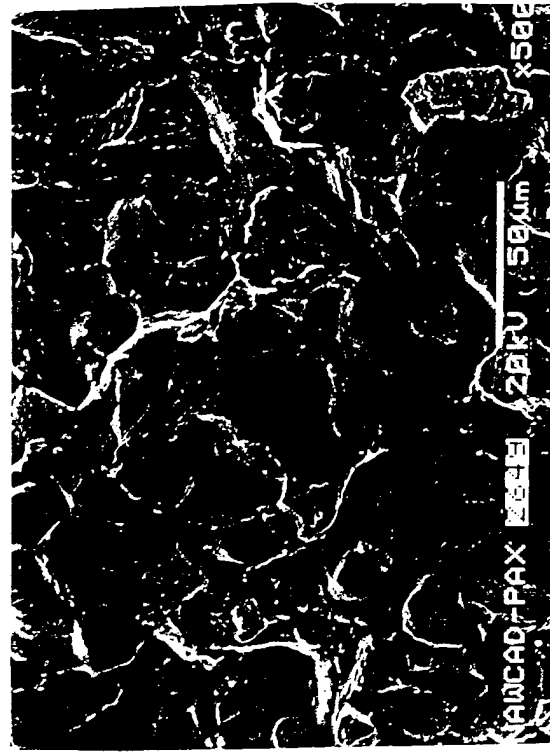
Figure 5. SEM Fractographs of AerMet 100 Steel, SCC Tested in Aqueous 3.5% NaCl Solution



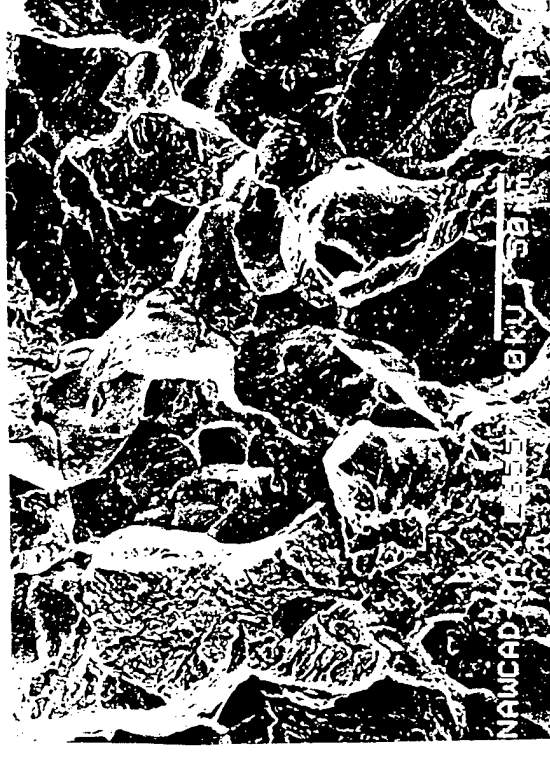
-1.2 V_{SCE}



-1.0 V_{SCE}



-0.8 V_{SCE}



-0.7 V_{SCE}

Figure 6. SEM Fractographs of 300M Steel, SCC Tested in Aqueous 3.5% NaCl Solution

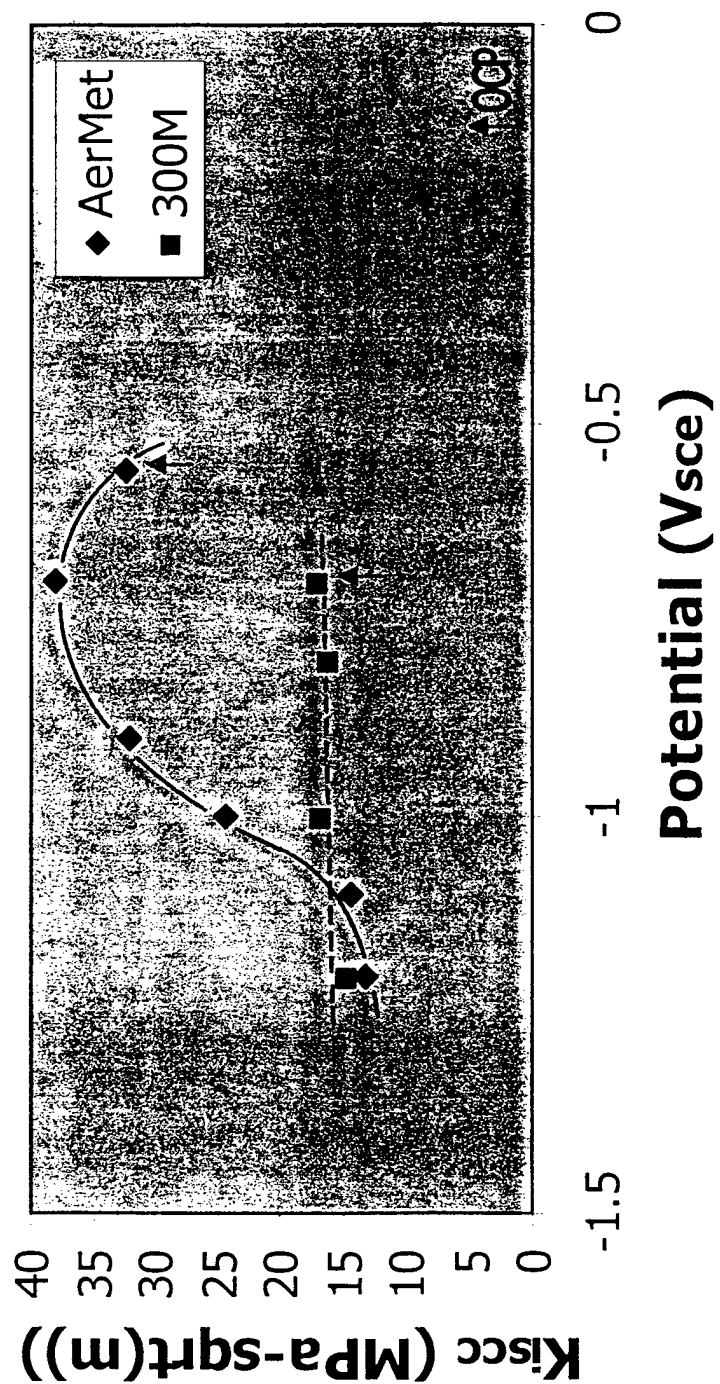


Figure 7. Variation of K_{isc} with Potential in Aqueous Solution of 3.5% NaCl and 2.8% Na_2SO_4

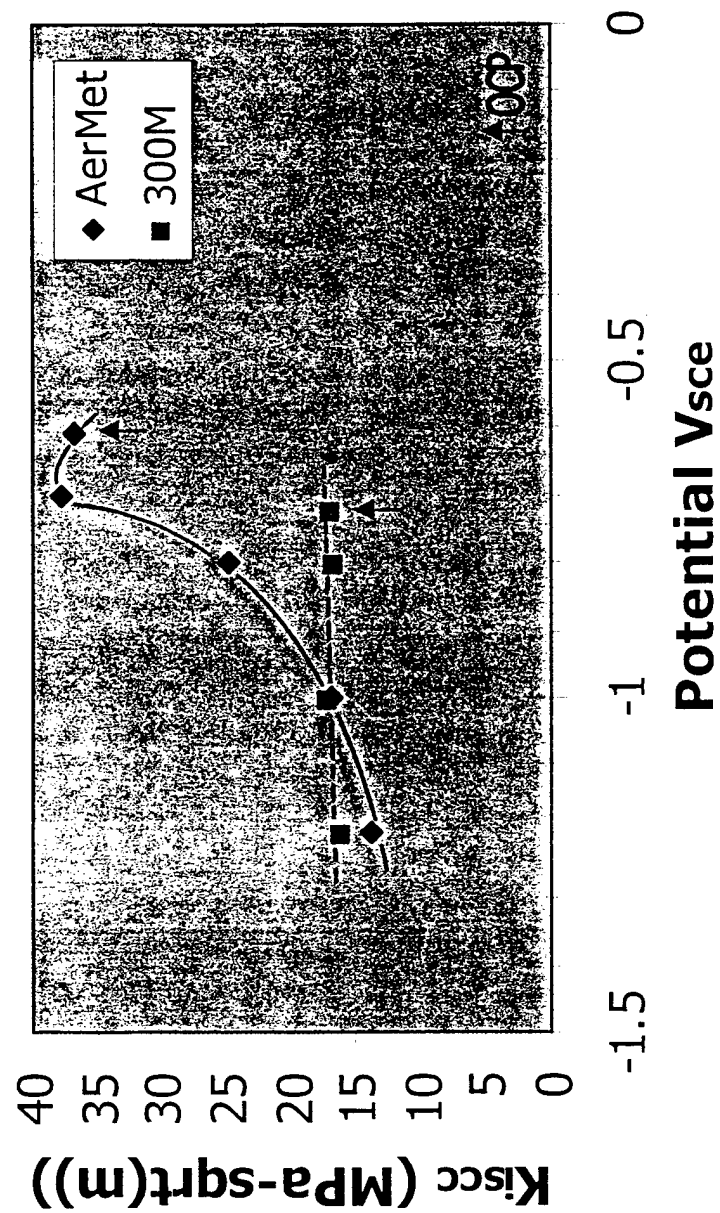


Figure 8. Variation of K_{ISCC} with Potential in Aqueous Solution of 0.35% NaCl and 6.5% Na_2SO_4

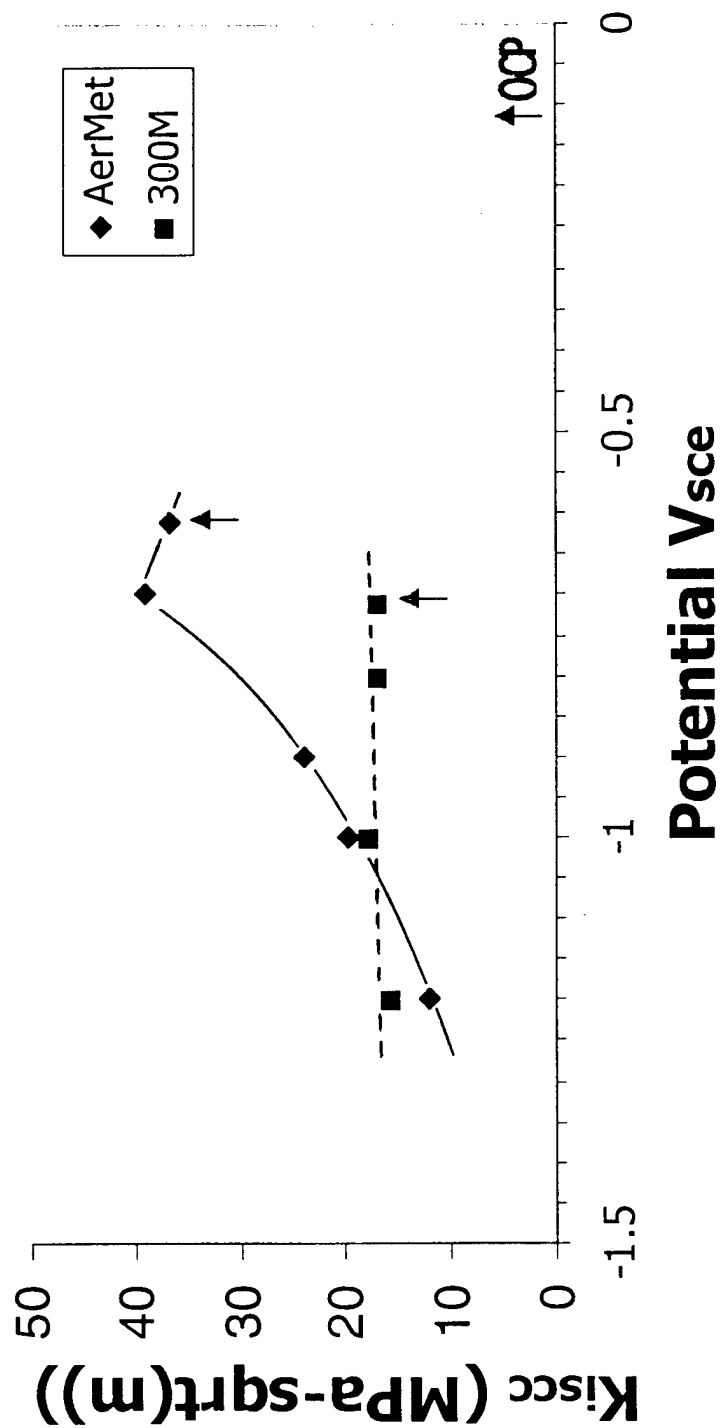


Figure 9. Variation of K_{ISCC} with Potential in Aqueous Solution of 0.035% NaCl and 6.9% Na_2SO_4